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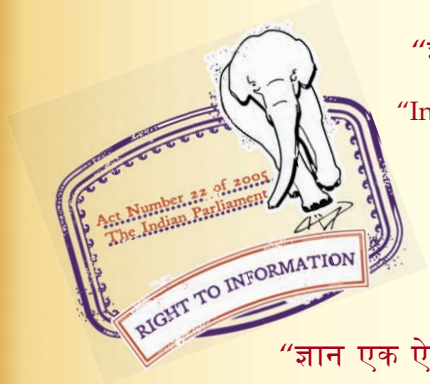
IS 328 (1992): Oil of eucalyptus globulus [PCD 18: Natural and Synthetic Fragrance Materials]



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"Knowledge is such a treasure which cannot be stolen"

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IS 328 : 1992
(Reaffirmed 2003)

भारतीय मानक

यूकेलिप्टस ग्लोबुलस का तेल — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

OIL OF EUCALYPTUS GLOBULUS — SPECIFICATION

(Second Revision)

First Reprint JUNE 2007
(Including Amendment 1)

UDC 665·526·78

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1992

Price Group 2

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was originally published in 1952 and first revised in 1957. In the original version, requirement for cineole content was prescribed as 55 percent minimum which was revised to 60 percent in 1957. Currently International Standard for the material prescribes minimum of 70 percent cineole content. Although cineole content of oil of Eucalyptus Globulus produced naturally in India is around 60 to 63 percent which is upgraded to as high as 70 percent or even higher by fractional distillation, the Committee responsible for formulation of this standard felt that limit of cineole content need not be raised further from the present value of 60 percent as the standard pertains to natural essential oil of *Eucalyptus globulus*, while the higher concentration of cineole content is obtained only after processing.

In this second revision an additional requirement for freezing point is being included. Other changes as necessary to bring the standard in line with current trade practices prevailing in the industry have also been made. While keeping *O*-cresol method of analysis for determination of citral content as referee method, GLC method of analysis has been included for guidance only.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off values should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 DECEMBER 1998
TO
IS 328 : 1992 OIL OF EUCALYPTUS GLOBULUS — SPECIFICATION

(Second Revision)

[*Page* 2, *Table* 1, *Sl No.* (v), *col* 3] — Substitute '1.456 1 - 1.466 9' *for* '1.455 9 to 1.466 7'.

(*Page* 4, *clause* D-1.1, *line* 10) — Substitute '3 m' *for* '2.43 m'.

(*Page* 4, *clause* D-2) — Delete.

(PCD 18)

Indian Standard

OIL OF EUCALYPTUS GLOBULUS — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for oil of *Eucalyptus globulus.*

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
323 : 1959	Rectified spirit (*revised*)
326	Methods of sampling and test for natural and synthetic perfumery materials:
(Part 1) : 1984	Sampling (*second revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (*second revision*)
(Part 3) : 1980	Relative density (*second revision*)
(Part 4) : 1980	Determination of optical rotation (*second revision*)
(Part 5) : 1986	Determination of refractive index (*second revision*)
(Part 6) : 1986	Determination of solubility in ethanol (*second revision*)
(Part 13) : 1981	Determination of cineole content (*second revision*)
(Part 18) : 1984	Determination of freezing point (*second revision*)
695 : 1986	Acetic acid (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials (*first revision*)
6597 : 1988	Glossary of terms relating to natural and synthetic perfumery materials (*first revision*)

3 TERMINOLOGY

For the purpose of this standard, definitions given under IS 6597 : 1988 shall apply.

4 SAMPLING

Representative samples of the material, shall be drawn as prescribed in IS 326 (Part 1) : 1984.

5 REQUIREMENTS

5.1 Description

The oil of eucalyptus shall be obtained by water or steam distillation of fresh leaves of *Eucalyptus globulus* Labill, or from other cineole-containing species of eucalyptus (family Myrtaceae) and rectified. The oil shall be a clear liquid, free from sediment, suspended matter, separated water and added adulterants.

5.2 Solubility

The oil of eucalyptus shall be soluble in an equal volume of ethyl alcohol, 80 percent by volume, when tested as prescribed in IS 326 (Part 6) : 1986.

5.3 Aldehydes

The oil shall satisfy the test requirements for the limit of aldehydes given in Annex A.

5.4 Phellandrene

The oil shall satisfy the requirements of the test for absence of phellandrene given in Annex B.

5.5 Reaction

A solution of recently distilled oil of eucalyptus in an equal volume of 80 percent by volume ethyl alcohol shall be neutral to moistened blue or red litmus paper.

5.6 The oil of eucalyptus shall also comply with the requirements given in Table 1.

6 TESTS

6.1 Tests shall be conducted as prescribed in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests, and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

2– –48 BIS/ND/07

Table 1 Requirements for Oil of *Eucalyptus Globulus*

(*Clause* 5.6)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Colour and appearance	Colourless or pale yellow liquid	IS 326 (Part 2) : 1980
ii)	Odour and taste	Characteristic aromatic camphoraceous sharp odour; followed by a sensation of cold	IS 2284 : 1988
iii)	Relative density at 27°/27°C	0·898 5 to 0·905 5	IS 326 (Part 3) : 1980
iv)	Optical rotation	− 5° to +10°	IS 326 (Part 4) : 1980
v)	Refractive index at 27°/27°C	1·455 9 to 1·466 7	IS 326 (Part 5) : 1986
vi)	Cineole content, per cent by mass, *Min*	60	IS 326 (Part 13) : 1981
vii)	Freezing point, °C	35 and above	Annex C

ANNEX A

(*Clause* 5.3)

DETECTION OF ALDEHYDES

A-1 REAGENTS

The following reagents are required.

A-1.1 Ethyl Alcohol

60 percent (by volume), obtained by diluting rectified spirit (conforming to IS 323 : 1959)

A-1.2 Standard Hydrochloric Acid — 0·5 N.

A-1.3 Methyl Orange Indicator

Prepare a 0·04 percent solution (w/v) of methyl orange in alcohol.

A-1.4 Standard Potassium Hydroxide Solution

Approximately 0·5 N. Prepare the standard solution in 60 percent alcohol and standardize against standard hydrochloric acid, using methyl orange indicator.

A-1.5 Hydroxylamine Hydrochloride Solution

Dissolve 35 g of hydroxylamine hydrochloride in 950 ml of ethyl alcohol and add about 3 ml of methyl orange indicator. Add standard potassium hydroxide solution until the full yellow colour of the indicator is obtained. Make up with ethyl alcohol to 1 000 ml.

A-1.6 Benzene

A-2 APPARATUS

A-2.1 Stoppered Tube

Approximately 150 mm in length and 25 mm in diameter.

A-3 PROCEDURE

A-3.1 Mix 10 ml of the material in the stoppered tube with 4 ml of hydroxylamine hydrochloride solution and add 5 ml of benzene. Add one drop of methyl orange indicator and titrate the liberated acid with standard potassium hydroxide solution. Shake vigorously for two minutes after each addition of standard potassium hydroxide solution and allow the layers to separate. Note the colour of the lower layer. The end point is reached when the full yellow colour of the indicator is permanent in the lower layer.

A-3.2 The limit prescribed shall be taken as not having been exceeded if the volume of standard potassium hydroxide solution (corrected to exactly 0·5 N) required for the titration does not exceed 2 ml.

ANNEX B

(*Clause* 5.4)

DETECTION OF PHELLANDRENE

B-1 REAGENTS

The following reagents are required.

B-1.1 Light Petroleum

Boiling range 50° to 60°C.

B-1.2 Sodium Nitrite Solution

A saturated solution of sodium nitrite in distilled water.

B-1.3 Glacial Acetic Acid

Conforming to IS 695 : 1986.

B-2 PROCEDURE

B-2.1 Mix 1 ml of the material with 5 ml of light petroleum and add to the solution 2 ml of sodium nitrite solution and then 2 ml of glacial acetic acid. Shake the mixture gently.

B-2.2 Phellandrene shall be taken to be absent if no crystalline precipitate forms in the upper layer in 10 minutes.

ANNEX C

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF FREEZING POINT

C-0 GENERAL

Cineole content in oil of *Eucalyptus globulus* may be determined by refering to a table within an accuracy of about ± 3 percent if the freezing congealing point is determined accurately. Cocking's *O*-cresol method is being prescribed here for determination of freezing point.

C-1 APPARATUS

C-1.1 Test Tube — stout walled about 95 mm diameter, 80 mm long.

C-1.2 Calibrated Thermometer — graduated in fifths of a degree.

C-1.3 Bottle — wide mouthed.

C-2 REAGENTS

C-2.1 Sodium Sulphate Anhydrous

C-2.2 *O*-Cresol — pure and dry, with a freezing point below 30°C. It is hygroscopic, therefore should be stored in small well stoppered bottle.

C-3 PROCEDURE

C-3.1 In a stout walled test tube (**C-1.1**) place about 3 g of accurately weighed oil sample, previously dried with anhydrous sodium sulphate. Add to it 2·1 g of melted *O*-cresol. Insert a thermometer (**C-1.2**) and stir the mixture well in order to induce crystallization. Note the highest reading of the thermometer.

C-3.1.1 Warm the tube gently until the contents are thoroughly melted and insert the tube through a bored cork into a wide mouthed bottle which is to act as an air jacket. The thermometer should be suspended from a ring stand in such a way that it does not touch the walls of the inner tube. Allow the mixture to cool slowly until crystallization commences, or until the temperature has fallen to the point previously noted. Stir the contents of the tube vigorously with the thermometer, rubbing the latter with the sides of the tube with an up and down motion in order to induce rapid crystallization.

C-3.1.1.1 Continue stirring and rubbing as long as the temperature rises. Take the highest point as the freezing point. Repeat this procedure until two readings agree within 0·1°C. Report the freezing point.

C-3.2 The percentage of cineole content may be computed from Table 1 of IS 326 (Part 13) : 1981.

ANNEX D

(Foreword)

DETERMINATION OF CINEOLE CONTENT BY GAS CHROMATOGRAPHIC METHOD

D-0 GENERAL

D-0.1 The chromatographic conditions given here are for guidance only.

D-0.2 Outline of the Method

A sample of the material is dissolved in a suitable solvent (for example, hexane, cyclohexane and petroleum ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

D-1 APPARATUS

D-1.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for oil of *Eucalyptus globulus* using a chromatograph with the following chromatographic conditions is shown in Fig. 1.

Column	
Material	Stainless Steel
Length	2·43 m
OD	0·32 cm
ID	0·20 cm
Stationary phase	FFAP*, 10 percent by mass
Solid support	Chromosorb WAW 60 to 80 mesh
Carrier Gas	Nitrogen
Conditions	
Column temperature isothermal	120°C
Injection port temperature	200°C
Detector	
Type	F. I. D.
Temperature	285°C

D-2 CALCULATION

D-2.1 Area Measurements (*see* Note 1)

Since normal peaks approximate a triangle, the area is measured by multiplying the peak height with the width of the half-height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

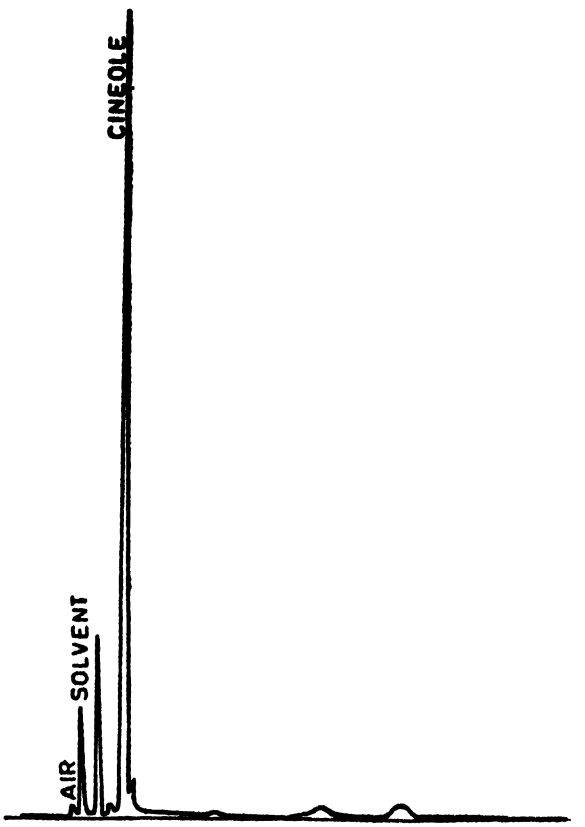

FIG. 1 A TYPICAL CHROMATOGRAM OF EUCALYPTUS OIL (GLOBULUS)

D-2.2 Area Normalization (*see* Note 2)

By normalizing, it is meant calculating the percentage composition by measuring the area of each peak and dividing the individual areas by total area, for example;

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTES

1 Other methods of area measurement, namely triangulation, disc integrator and electronic digital integrator, if fixed with GLC machine, would be of great advantage.

2 Internal standardization may be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

*Free fatty acid phase (FFAP) in carbowax 20M treated with nitrophthalic acid.

GMGIPN—48 BIS/ND/07—100

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc : No. PCD 18 (1056).

Amendments Issued Since Publication

Amendment No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.org.in

Regional Offices:		Telephones
Central :	Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110 002	2323 7617 2323 3841
Eastern :	1/14, C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi KOLKATA 700 054	2337 8499, 2337 8561 2337 8626, 2337 9120
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